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(56) Documents Cited

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US 4235676 A

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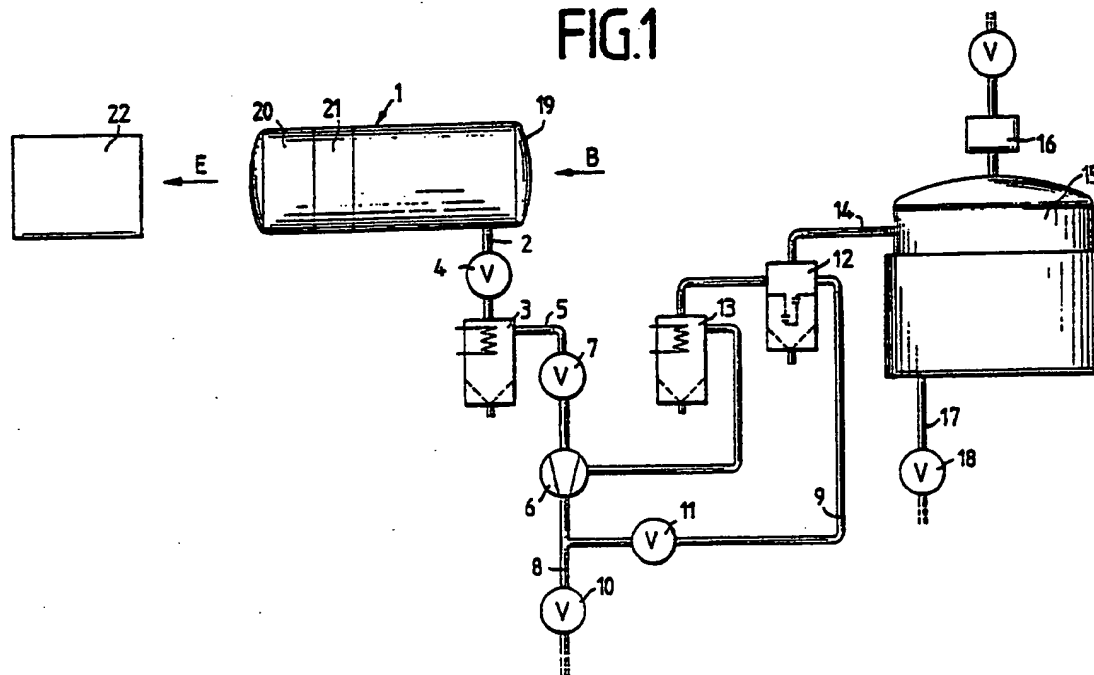
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(54) Treating waste plastic or rubber

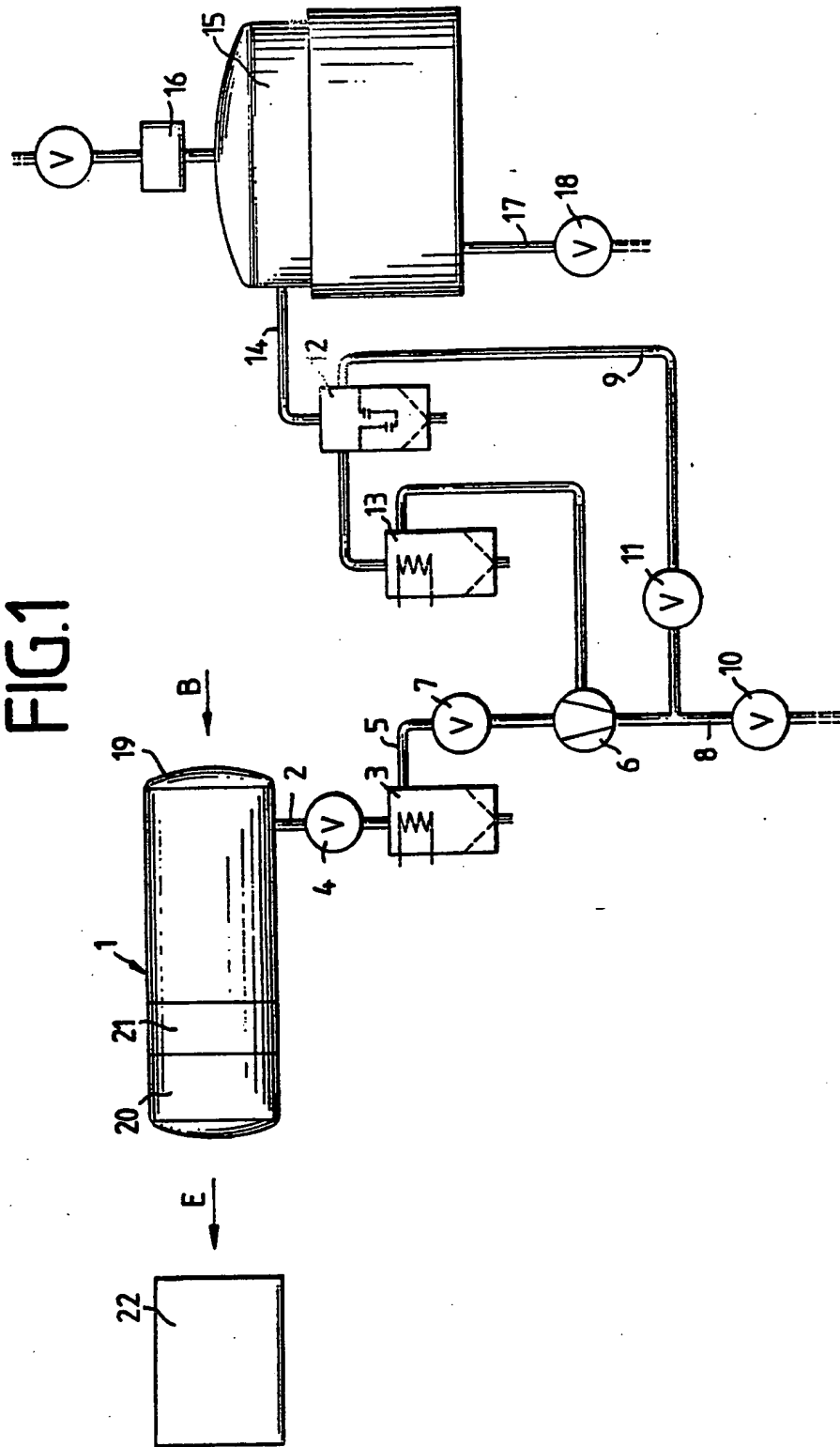
(57) Process and device for treating articles of plastic or rubber, in which the articles are placed in a process chamber (1) for embrittlement, the chamber is evacuated and heated above 250°C and the articles are then mechanically crushed and the vapours and gases eg of metals, greases or oils arising in the chamber are led out into an essentially closed system which comprises in particular pipes, condenser, pumps and reservoir and in which both the embrittled and crushed articles and the vapours and gases are conveyed for either reprocessing or controlled disposal. The crushed residue is a carbonaceous material.

FIG.1



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FIG.1



Process and device for treating articles essentially comprising plastic or rubber

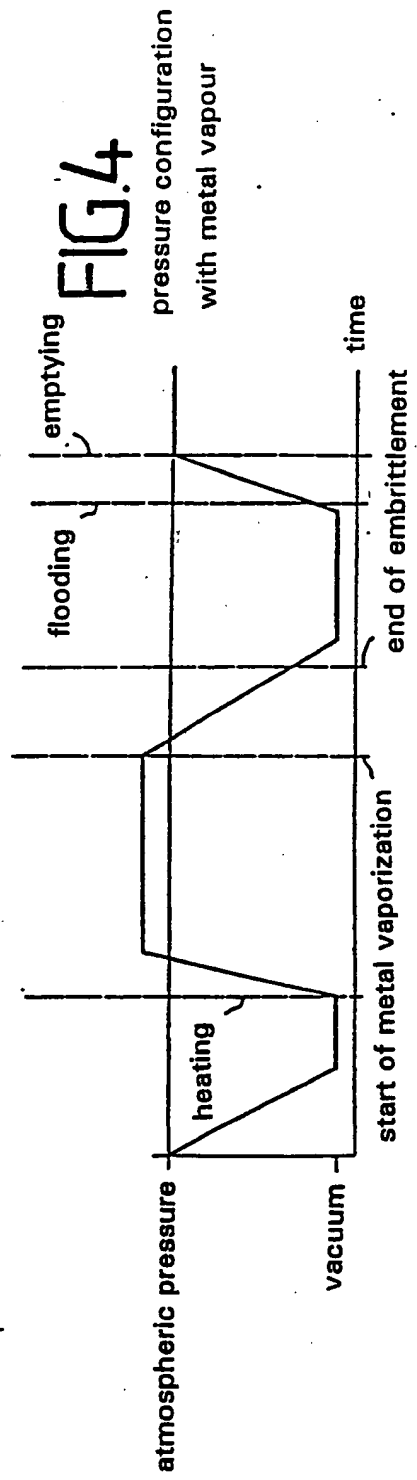
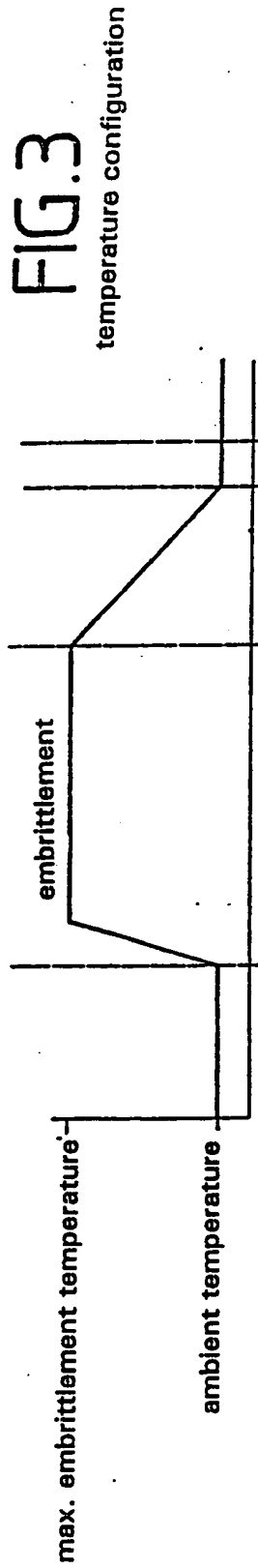
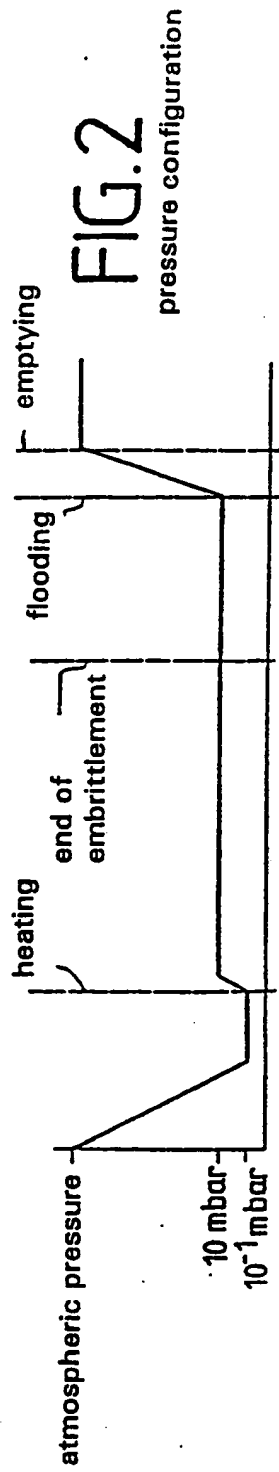
The invention relates to a process and a device for embrittling and crushing articles essentially comprising plastic or rubber, such as boots or seals.

Enormous amounts of waste containing plastic or rubber are generated in both the private and the industrial field. It is becoming increasingly difficult to dispose of this waste for ecological reasons. The only economic process known to date is burning, but this is not without problems because chlorine and sulphur compounds are released to some extent. Rubber waste to some extent contains metal inserts with anti-corrosion (e.g. Zn) supports which are oxidized and released as fine dust. Very large quantities of air are used in the combustion processes and these have to be scrubbed in large gas and dust filters.

Recycling processes for the valuable materials that are produced in this case have not yet been considered. Drive shafts in motor vehicles, for example, are protected from contamination and dust with flexible rubber boots. In service these boots have a two-fold function. Firstly they represent a reservoir provided with lubricant for the joint and secondly they prevent the ingress of water and corrosive contamination.

These boots need to be replaced after a limited service life due to ageing or through damage. This virtually always takes place when the drive shaft is replaced. The worn boots are designated as problem waste. When cars are scrapped, these boots also occur along with other components such as shaft seals, packings, O-rings or other seals.

Reprocessing or even simply disposal would be possible if the plastic or rubber were separated from the lubricants, for



which washing processes have mostly been used in the past. A further process for removing grease and oil from components containing plastic or rubber is thermal vaporization of the greases or oils in a vacuum. A process for this is already known in the prior art (DE P 4136990.4). This document describes a process for degreasing and cleaning articles affected by substances containing grease and/or oil and is characterized in that the articles to be cleaned are heated in a process chamber under vacuum conditions in such a way that the greases and/or oils can be separated from the articles according to their physical condition.

The rubber components degreased in this way must be cut into small pieces and ground for further processing purposes. The grinding process is much more difficult than the degreasing although it can be carried out by undercooling with, for example, liquid nitrogen.

The disadvantage of these known processes and devices is, for example, that even after combustion large quantities of solid and gaseous waste materials still remain, which are either disposed of separately or to some extent released into the atmosphere also through combustion. The waste gases from combustion contain large quantities of metal oxides (e.g. zinc, cadmium, vanadium, arsenic oxide etc.), some of which are highly toxic.

The object of the invention is to propose a process and a device so that on the one hand the articles to be treated are conditioned so that they can subsequently be crushed without problems and that on the other hand at the same time, the quantity of gas produced when plastic or rubber is disposed of is minimized and fed in a closed gas circuit and both the gases and the solids can be conveyed for reprocessing or disposal that is ecologically and financially acceptable.

This object is achieved according to the invention in that on the one hand, the articles degreased according to a known process are placed in a process chamber, evacuated and embrittled by heating to a temperature above 250 °C and on the other hand the gases arising in the chamber are fed from the chamber in a closed gas circuit and the articles and the gases are then conveyed for reprocessing or disposal. When the heating process is completed the components embrittled in this way are mechanically crushed.

The lubricants distilled off by the known process can then be reprocessed and conveyed for re-use. If the components cleaned in this way remain in the vacuum process chamber after degreasing and if the process temperature is further increased, these originally flexible plastic or rubber components lose their elasticity. The organic compounds are destroyed and a carbon skeleton remains. The readily vaporizable metals (such as Zn and Cd) are separated by metallic means in the condenser. The metal condensate settles at the bottom of the condenser because of the higher density compared with the oils. This carbon skeleton - free from vaporizable metals - can then be crushed or ground to powder without problems. The S, Cl, F and N compounds which occur in the embrittlement process can be neutralized and precipitated according to known methods.

Compared with traditional combustion, in the process according to the invention, advantageously only substantially smaller quantities of waste gas have to be treated. At the same time this process can be carried out as a closed process so that no gases can inadvertently escape into the ambient air. In contrast the gases collected in the gas container can be determined by means of a gas analyzer and conveyed for specific re-use or disposal.

This can be achieved, for example, by placing the gases in bottles that can be transported.

When ground to powder for example, the embrittled rubber components can be used wherever pulverized coal or coal dust is used and reprocessed into coal or graphite semi-finished products, possibly into rubber articles again. All distillation products can be processed into new lubricants.

In an embodiment, oil-contaminated rubber components were heated in a vacuum degreasing installation to initially 200 °C and then to 300 °C in order to embrittle them. The process time for the heating from 20 °C ambient temperature to 300 °C was approx. three hours. When 300 °C were reached the installation was switched off and cooled at natural speed. Plastic and rubber articles of any kind can, for example, be embrittled according to this process and with this device. The articles in mind here are, for example, car tyres, boots, seals, bags, cups and other mouldings.

Further embodiments and features are described in greater detail and characterized in the sub-claims.

A wide variety of embodiments is possible with the invention; one of these is described in greater detail in the accompanying drawings in which :

- Fig. 1 shows a diagrammatic view of the process components for a rubber embrittlement process,
- Fig. 2 shows the pressure configuration in the course of an embrittlement process,
- Fig. 3 shows the temperature configuration in the course of an embrittlement process,
- Fig. 4 shows the pressure configuration in the course of an embrittlement process with simultaneous metal vapour condensation.

An electrically heated or gas/oil-fired process chamber 1 is connected to a condensate separator 3 via a condensate line 2. A shut-off valve 4 is provided in the line 2. The separator 3 is connected to a vacuum pump 6 via a suction line 5, a valve 7 being arranged in the suction line 5.

On the outlet side of the vacuum pump 6 is a discharge line 8 and a ring line 9 both of which can be controlled by the valves 10 and 11. A gas neutralizer 12 and a gas dryer 13 are also provided in the ring line 9. A line 14 which connects the neutralizer 12 to a gas container 15 is arranged on the neutralizer 12.

On the gas container 15 is a gas analyzer 16 as well as a discharge line 17 which can be closed by means of the valve 18 and can, for example, be used for putting the gas into transport bottles.

The process chamber 1 is designed as a sluice chamber so that the articles to be treated are placed in the chamber 1 via a loading door 19 on one side of the chamber and can be removed from the chamber 1 via a discharge door 20 on the opposite side of the chamber when the treatment process is completed. The discharge door 20 can be separated from the treatment compartment of the chamber 1 by means of a sluice 21.

Adjacent to the process chamber 1 in the direction of discharge E is a silo 22 or a cooling device as an intermediate store for a grinding device which is not shown and in which the embrittled articles are subsequently mechanically crushed and compressed if required.

Figs. 2, 3 and 4 show examples of the pressure and temperature configuration over time inside the process chamber 1 in the course of an embrittlement process.



After the initially flexible, soft articles have been placed in the chamber, starting from atmospheric pressure (Fig. 2) a vacuum of  $10^{-1}$  mbars, for example, is initially set and kept constant for a short time. The heating process now begins so that the chamber pressure increases slightly, to 10 mbars for example. Provided that the process-chamber is continuously evacuated, this pressure remains virtually constant beyond the end of the embrittlement process and until the flooding of the chamber begins. The chamber pressure then increases again to atmospheric pressure and the chamber can be opened and emptied.

The temperature configuration (Fig. 3) remains constantly at ambient temperature until the heating process begins. Heating now continues steadily up to embrittlement temperature; this usually begins at 250 °C for the rubber workpieces to be treated and extends to over 400 °C. If the workpieces to be embrittled do not, however, consist exclusively of rubber, plastic or the like, but still contain metal inserts, as is the case with steel radial tyres, for example, the process temperature must be above the vaporization temperature of the metal in question. For cadmium this is at least 320 °C, for example, and at least 400 °C for zinc, at a metal vapour pressure  $> 10^{-1}$  mbars in each case.

When the embrittlement process has been completed the batch is centrifuged or the chamber temperature is cooled down to ambient temperature again and the chamber is flooded and emptied at this temperature.

Compared to the pressure configuration shown in Fig. 2, the pressure adopts a different configuration (Fig. 4) when metal is also vaporized from the articles in the course of embrittlement, as has already been mentioned in respect of the temperature profile in Fig. 3. In this case the pressure changes (in the course of actual embrittlement) because when

heating commences the process chamber is sealed and no longer pumped off. The chamber pressure thus rises to a value that may be above atmospheric pressure. When metal vaporization begins the suction line to the chamber is opened again, the chamber is evacuated again and the pressure drops to its minimum value until after embrittlement has finished. When the chamber is completely evacuated the vacuum pressure is kept at the minimum value for a short time longer and the chamber is then flooded and emptied.

## CLAIMS

1. Process for treating articles essentially comprising plastic or rubber, in which on the one hand the articles are placed in a process chamber (1) for embrittlement, the chamber (1) is evacuated and heated and the articles are then mechanically crushed and in which on the other hand the vapours and gases arising in the process chamber (1) are led out of the chamber (1) into an essentially closed system which comprises in particular pipes, condenser, pumps and reservoir and in which both the embrittled and crushed articles and the vapours and gases are conveyed for either reprocessing or controlled disposal.
2. Process according to Claim 1, characterized in that before embrittlement the grease and/or oil contaminated articles are degreased and/or de-oiled in a pre-cleaning step by means of heating in a vacuum.
3. Process according to Claim 2, characterized in that the temperature or the pressure in the pre-cleaning step is lower than the temperature or the pressure in the course of embrittlement.
4. Process according to Claim 1 or 3, characterized in that the temperature in the process chamber (1) in the course of embrittlement is set to at least 250 °C for a time.
5. Process according to Claim 4, characterized in that the process chamber (1) is heated inductively, by electric resistance heating or by direct firing.
6. Process according to Claim 1, characterized in that the pressure in the process chamber (1) in the course of embrittlement is set below atmospheric pressure.

7. Process according to Claim 6, characterized in that the pressure in the process chamber (1) is set below 10 mbars for a time.
8. Process according to Claim 1, characterized in that to prevent the vaporization of metallic constituents in the articles the pressure in the process chamber (1) is set above atmospheric pressure for a time.
9. Process according to Claim 1, 2 or 3, characterized in that the degreasing and embrittlement of the articles to be treated can be carried out in one and the same process chamber (1).
10. Process according to one or more of the preceding Claims, characterized in that the embrittled articles are conveyed to a grinding process and are then compressed.
11. Device for embrittling and crushing articles essentially comprising plastic or rubber by means of a thermal vacuum and a mechanical crushing process, essentially comprising a vacuum process chamber (1), at least a condensate separator (3), a vacuum pump (6), a gas neutralizer (12), a gas dryer (13) and a gas container (15) with a gas analyzer (16) and several valves (7, 10, 11, 18), a sluice (18), a silo (19) or a cooling chamber and a grinding device.
12. Device according to Claim 11, characterized in that the process chamber (1) can be separated from the other components by means of at least one valve (4).
13. Device according to Claim 12, characterized in that the valve (4) can be designed as a control valve.

14. Device according to Claims 11, 12 or 13, characterized in that a condenser is provided as an oil vapour condenser.
15. Device according to Claims 11, 12 or 13, characterized in that a condenser is provided as a metal vapour condenser.
16. Device according to one or more of Claims 11 to 15, characterized in that the gases are initially collected in the gas container (15).
17. Device according to Claim 16, characterized in that the gases can be discharged from the gas container (15) separately.
18. Device according to Claim 16, characterized in that nitrogen ( $N_2$ ) can be discharged into the ambient air.
19. Device according to Claim 16, characterized in that hydrogen ( $H_2$ ) is subsequently combusted.
20. Device according to Claim 11, characterized in that the process chamber (1) is provided as a sluice chamber with a loading and a discharge opening.
21. Device according to Claim 11, characterized in that a silo or a cooling chamber and a grinding device are connected to the process chamber (1).
22. Device according to Claim 21, characterized in that a sluice is provided between process chamber (1) and silo or cooling chamber.

## LIST OF REFERENCE NUMERALS

- 1 Process chamber
- 2 Condensate line
- 3 Condensate separator
- 4 Valve
- 5 Suction line
- 6 Vacuum pump
- 7 Valve
- 8 Discharge line
- 9 Ring line
- 10 Valve
- 11 Valve
- 12 Gas neutralizer
- 13 Gas dryer
- 14 Line
- 15 Gas container
- 16 Gas analyzer
- 17 Discharge line
- 18 Valve
- 19 Loading door
- 20 Discharge door
- 21 Sluice
- 22 Silo
  
- B Direction of loading
- E Direction of discharge

Relevant Technical Fields

- (i) UK Cl (Ed.M) C5E (EBC, EDH, EDM, EF)  
(ii) Int Cl (Ed.5) C10B 53/00; C10G (1/02, 1/10)

Search Examiner  
R E HARDY

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22 JUNE 1994

Databases (see below)

(i) UK Patent Office collections of GB, EP, WO and US patent specifications.

Documents considered relevant following a search in respect of Claims :-  
1-10

(ii) WPI

Categories of documents

- Document indicating lack of novelty or of inventive step. P: Document published on or after the declared priority date but before the filing date of the present application.
- Document indicating lack of inventive step if combined with one or more other documents of the same category. E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.
- Document indicating technological background and/or state of the art. &: Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages		Relevant to claim(s)
Y1	GB 2228493 A	(GUANGDONG) See page 3 lines 27-29 and Examples	Claim 1 at least
X, Y1, Y2	GB 1547962 A	(DECO) See Figure	Claim 1 at least
Y2	GB 1494300 A	(OIL SHALE) Note grinding during pyrolysis	Claim 1 at least
Y2	GB 1441997 A	(INDUSTRIAL SCIENCE) Note pulverising step	Claim 1 at least
X, Y1, Y2	EP 0426926 A1	(RING OIL) Figures 1-4	Claim 1 at least
X, Y1, Y2	WO 93/00449 A1	(LAVAL) See the Figure	Claim 1 at least
X, Y1, Y2	WO 93/12198 A1	(JARREL) See the Figure and note final chav is crushed	Claim 1 at least
Y1	US 4740270 A	(ROY) Column 3 lines 59-65	Claim 1 at least
X, Y1, Y2	US 4235676 A	(DECO) See Figure	Claim 1 at least

Databases: The UK Patent Office database comprises classified collections of GB, EP, WO and US patent specifications as outlined periodically in the Official Journal (Patents). The on-line databases considered for search are also listed periodically in the Official Journal (Patents).

## Relevant Technical Fields

- (i) UK Cl (Ed.M) C5E (EAD EBC EDH)  
(ii) Int Cl (Ed.5) C10B (53/00, 02, 04, 06); C10G (1/02, 10)

Search Examiner  
R E HARDYDate of completion of Search  
27 OCTOBER 1994

## Databases (see below)

(i) UK Patent Office collections of GB, EP, WO and US patent specifications.

Documents considered relevant following a search in respect of Claims :-  
11-22

(ii) WPI

## Categories of documents

- X: Document indicating lack of novelty or of inventive step. P: Document published on or after the declared priority date but before the filing date of the present application.
- Y: Document indicating lack of inventive step if combined with one or more other documents of the same category. E: Patent document published on or after, but with priority date earlier than, the filing date of the present application.
- A: Document indicating technological background and/or state of the art. &: Member of the same patent family; corresponding document.

Category	Identity of document and relevant passages		Relevant to claim(s)
Y	GB 1547962 A	(DECO) See the Figures	Claim 11 at least
Y	GB 1531106 A	(LAMPL) See the Figures	Claim 11 at least
Y	EP 0003803 A1	(INTENCO) See the Figures and Examples	Claim 11 at least
Y	US 5138959 A	(KULKARNI) See Figure 3, ref 68	Claim 11 at least
Y	US 5057189 A	(APFFEL) See Figure 1A refs 28 and 31	Claim 11 at least
Y	US 4235676 A	(DECO) See the Figures	Claim 11 at least
Y	WO 93/12198 A1	(JARRELL) See pages 12-13 and the Example	Claim 11 at least
Y	WO 93/00449 A1	(LAVAL) See the Figure	Claim 11 at least

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